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ER Document Catalog Number: <b>ER1999-0203</b>			
Author: <b>Bart J. Vanden Plas</b>			

Environmental Restoration Project  
Standard Operating Procedure

for:

# Routine Validation of High Explosives Data

**Los Alamos**

NATIONAL LABORATORY

Los Alamos, New Mexico 87545

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# Routine Validation of High Explosives Data

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### **List of Acronyms and Abbreviations**

ER	environmental restoration	MDL	method detection limit
SOP	standard operating procedure	%R	percent recovery
HE	high explosive	RN	request number
LANL	Los Alamos National Laboratory	COC	chain of custody
USTHAMA	US Army Toxic and Hazardous Materials Agency	FSF	Field Support Facility
SOW	statement of work	QC	quality control
EPA	US Environmental Protection Agency	SMO	Sample Management Office
		RPF	Records Processing Facility
EQL	estimated quantitation limit		
LCS	laboratory control sample	LAL	upper acceptance limit
CLP	Contract Laboratory Program	UAL	upper acceptance limit

# Routine Validation of High Explosives Data

**NOTE:** Environmental Restoration (ER) Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at <http://erinternal.lanl.gov/documents/Procedures/sops.htm>. However, it is their responsibility to ensure that they are trained to and use the current version of this procedure. Contact the author if text is unclear.

## 1.0 PURPOSE

This standard operating procedure (SOP) represents the minimum standard for evaluating high explosives (HE) analytical data. These data can be generated for the Los Alamos National Laboratory (LANL) ER Project using SW-846 Method 8330 or the comparable US Army Toxic and Hazardous Materials Agency (USATHAMA) method under the current statement of work (SOW) for analytical services (LANL 1995). The evaluation of data by this procedure is not specific to a particular data use, although this procedure may be used as a point of departure for developing focused data validation requirements specific to a particular data use.

**Note:** Implementation of this procedure will result in a tabulation of data compliances and noncompliances identified relative to expectations for data quality based on national guidelines for data review (EPA 1994). Because the acceptance criteria used for this procedure are not based on site-specific acceptance criteria, the results of this validation procedure are intended to be used as *general indicators* of data quality and should not be construed as a definitive identification of data usability.

**Note:** Implementation of this procedure may be followed by a more focused and data-use-specific evaluation of data, especially if implementation of this SOP indicates that the data may contain technical deficiencies.

## 2.0 TRAINING

All data validators who implement this SOP shall possess a minimum of a bachelors degree in chemistry and two years of experience in generating analytical data in an environmental analytical laboratory or two years' data validation experience. New validators shall work under the direct supervision of an experienced ER Project validator. The work of new validators shall be reviewed and signed by an experienced ER Project validator until ten data record packages for each analytical suite have been satisfactorily validated. ER Project validators shall have demonstrated familiarity with the US Environmental Protection Agency (EPA) national functional guidelines for data review. All data validators must

document that they have read and understand this SOP and completed all applicable training assignments in accordance with QP-2.2.

### 3.0 DEFINITIONS

- 3.1 Daily calibration — Combination of calibration blank and check standards used to determine if the instrument response to analyte concentration is within acceptable bounds relative to the initial calibration. A daily calibration is performed every 24 hrs of operation and establishes the 24-hr relative response factors on which quantitations are based, thus verifying the satisfactory performance of an instrument on a day-to-day basis. The daily calibration 24-hr period assumes that the liquid chromatograph has not been shut down during the 24-hr period.
- 3.2 Data validator — Person who has met the minimum standards of training established in Section 2.0 and who implements this SOP on behalf of the ER Project.
- 3.3 Estimated quantitation limit (EQL) — Lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The low point on a calibration curve should reflect this quantitation limit. The EQL is not used to establish detection status. See the SOW for analytical services (RFP No. 9-XS1-Q4257) for a more complete definition.
- 3.4 Holding time — Maximum time between sample collection and sample preparation and/or analysis that a sample can be stored without unacceptable changes in analyte concentrations. Holding times apply under prescribed storage conditions; deviations in storage conditions may affect holding times. Appropriate storage conditions for samples of various matrices scheduled for selected analyses may be found in the current LANL-ER-SOP-01.02, the applicable analytical method, and the current ER Project SOW for analytical services.
- 3.5 Initial calibration — Process used to establish the relationship between instrument response and analyte concentration at several analyte-concentration values to demonstrate that an instrument is capable of acceptable analytical performance. The initial calibration for HE analyses is performed at the beginning of each analytical sequence or as necessary if the continuing calibration acceptance criteria are not satisfied.
- 3.6 Laboratory control sample (LCS) — A known matrix that has been spiked with compound(s) representative of the target analytes. The LCS is used to document laboratory performance. The acceptance criteria for LCSs are method specific.

- 3.7 Laboratory duplicate sample — The portions of a sample taken from the same sample container, prepared for analysis and analyzed independently but under identical conditions; used to assess or demonstrate acceptable laboratory method precision at the time of analysis. Each duplicate sample is expected to be equally representative of the original material. Duplicate analyses also are performed to generate data, to determine the long-term precision of an analytical method on various matrices.
- 3.8 Laboratory qualifier (or laboratory flag) — Codes applied to the data by the contract analytical laboratory to indicate, on a gross scale, a verifiable or potential data deficiency. These flags are applied using the EPA CLP guidelines.
- 3.9 LANL data validation qualifiers — The data qualifiers defined by LANL and used in the ER Project baseline-validation process. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate ER Project SOP (ER-SOPs 15.01–15.06).
- 3.10 LANL data validation reason codes — The codes applied to the sample data by data validators who are independent of the contract laboratory which performed the sample analysis. Reason codes provide an in-depth and analysis-specific explanation for applying the qualifier with some description of the potential impact on the data use. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate ER Project SOP (ER-SOPs 15.01–15.06).
- 3.11 Method blank — Analyte-free matrix to which all reagents are added in the same volumes or proportions as those used in the environmental sample processing, and which is prepared and analyzed in the same manner as the corresponding environmental samples. A method blank is used to assess the potential for sample contamination during preparation and analysis.
- 3.12 Method detection limit (MDL) — Minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined from analysis of samples of a given matrix type that contain the analyte after subjecting the sample to the usual preparation and analyses. The MDL is used to establish detection status.
- 3.13 Percent recovery (%R) — Amount of material detected in a sample (minus any amount already in the sample) divided by the amount added to the sample and expressed as a percentage.
- 3.14 Request number (RN) — An identifying number assigned by the ER Project to a group of samples that are submitted for analysis.
- 3.15 Routine data — Data generated using analytical methods that are identified as routine methods in the current ER Project SOW for analytical services.

- 3.16 *Routine data validation* — Process of reviewing analytical data relative to quantitative routine acceptance criteria. The objectives of routine data validation are to (1) estimate the data's technical quality relative to minimum national guidelines adopted by the ER Project, and (2) indicate to data users the technical data quality at a general level by assigning qualifier flags to environmental data whose quality indicators do not meet acceptance criteria.
- 3.17 *Surrogate compound (surrogate)* — Organic chemical compound used in the analyses of organic target analytes that is similar in composition and behavior to target analytes but is not normally found in environmental samples. Surrogates are added to every blank, sample, and spike to evaluate the efficiency with which analytes are recovered during extraction and analysis.
- 3.18 *Target analyte* — An element, chemical, or parameter, the concentration, mass, or magnitude of which is designed to be quantified by use of a particular test method.

## 4.0 BACKGROUND AND PRECAUTIONS

- 4.1 To protect the integrity of the data record package, the **data validator** must store and handle all data record packages under ER Project chain-of-custody (COC) rules prescribed in ER-SOP-15.09.
- 4.2 Logic diagrams are included in this SOP to expedite the validation process. Logic diagrams in this SOP do not include instructions about where to record validation results. Those instructions are incorporated in the text that corresponds to each logic diagram.
- 4.3 The HE data validation checklist forms identify actions that must be taken, depending on whether a validation condition is true or false (Attachment D). Look at the top of each validation form to learn the required action.
- 4.4 This validation process requires that the **validator** record qualifier flags and reason codes on photocopies of the data summary results forms (Form I) in the hard copy data record packages. Contiguous lines of identical qualification on the photocopied Form I may be represented as the qualifier flag and reason code, followed by a vertical downward arrow to the end of the block of results that are qualified identically.
- 4.5 The HE data validation checklist forms in Attachment D are examples of the forms the validator must use to validate data under this SOP. The forms may be reproduced in whole or in part, as needed to complete the validation of a data record package.

## 5.0 EQUIPMENT

The **validator** may need the following equipment and supplies to implement this procedure:

- 5.1 current HE data validation checklist forms (see Attachment D),
- 5.2 data record packages to be validated,
- 5.3 electronic calculator (optional),
- 5.4 photocopier, and
- 5.5 current ER Project SOW for analytical services.

## 6.0 PROCEDURE

**Note:** Deviations from SOPs are made in accordance with QP-4.2.

### 6.1 Prepare for Data Validation

- 6.1.1 The **validator** will begin by obtaining the required current versions of the HE data validation checklist forms (see Attachment D) from the ER Project website (<http://erinternal.lanl.gov/Quality/forms.htm>).
- 6.1.2 Obtain from the Sample Management Office (SMO) of the Field Support Facility the data record package(s) that contain the sample data to be validated.
- 6.1.3 Prepare a data validation cover sheet (see Attachment C) by completing the top part of the form and placing a check or other mark adjacent to the analytical suites for which this validation is being performed.

**Note:** You may use a single cover sheet when validating multiple analytical suites under the same RN.

**Note:** Use a separate sheet of paper to document each deficiency identified beyond the scope of this procedure, including phone conversations with the analytical laboratory personnel concerning these deficiencies. Attach these sheets to the data validation cover sheet.

- 6.1.4 Verify that the following items are present in each data record package:
  - 6.1.4.1 signed LANL COC record,
  - 6.1.4.2 case narrative,
  - 6.1.4.3 results forms (CLP Form I or equivalent) for each sample,
  - 6.1.4.4 chromatograms for each sample,
  - 6.1.4.5 chromatograms for standards,

- 6.1.4.6 quantitation reports, and
- 6.1.4.7 quality control (QC) forms for water and/or soils, as appropriate.
- 6.1.5 If the data record package does not contain all items listed in Sections 6.1.4.1 through 6.1.4.7, contact the FSF or the analytical laboratory to obtain those materials.
  - 6.1.5.1 If required documentation is missing from the data record package, and the package is less than six months old, contact the analytical laboratory and allow three business days for the laboratory to submit the required documentation.
  - 6.1.5.2 If the analytical laboratory does not submit documentation within three business days, return the data record package to the SMO for contract-compliance action.
  - 6.1.5.3 If the data record package is greater than 6 months old, allow 10 business days for the analytical laboratory to submit the required documentation before returning the data record package to the SMO.
- 6.1.6 Record the presence or absence (“Y” or “N”) of each item, as appropriate, in the completeness checksheet of the validation cover sheet.
- 6.1.7 In the data validation cover sheet completeness section, note any samples whose data are missing from the data record package.
- 6.1.8 Photocopy all analytical laboratory QC forms from the data record package.
- 6.1.9 Photocopy the case narrative from the data record package.
- 6.1.10 Photocopy the forms (Form I) that you will use during the validation process before completing the form.

**Caution:** Do not record data-validation qualifiers and reason codes on the original Form I.

**Note:** The **validator** must submit photocopies of the items listed in Sections 6.1.8 through 6.1.10 as attachments to the completed data validation forms.

## 6.2 Verify Initial Calibration

- 6.2.1 If an initial calibration *was completed*,
  - 6.2.1.1 record “Y” in block 1a of the HE data validation checklist, Part I;

- 6.2.1.2 record “n/a” in block 1c of the HE data validation checklist, Part I; and
- 6.2.1.3 go to Section 6.3, Verify Daily Calibration.
- 6.2.2 If the initial calibration *was not completed*,
  - 6.2.2.1 record “N” in block 1a of the HE data validation checklist, Part I;
  - 6.2.2.2 circle “A, H16” in block 2b of the HE data validation checklist, Part I;
  - 6.2.2.3 record the qualifier flag reason and code combination “A, H16” next to the results of all affected samples, on Form I; and
  - 6.2.2.4 record the time elapsed (to the nearest minute) completion of the initial calibration and affected samples in block 1c of the HE data validation checklist, Part I.

### 6.3 Verify Daily Calibration

**Note:** This validation check is not required if all samples were analyzed within 24 hrs of the initial calibration.

- 6.3.1 If a daily calibration *was performed* on the same day as, or within 24 hr of, the sample analyses,
  - 6.3.1.1 record “Y” in block 2a of the HE data validation checklist, Part I;
  - 6.3.1.2 record “n/a” in block 2c of the HE data validation checklist, Part I; and
  - 6.3.1.3 go to Section 6.4, Verify Method-Blank Results.
- 6.3.2 If a daily calibration *was not performed* on the same day as, or within 24 hrs of, the sample analyses,
  - 6.3.2.1 record “N” in block 2a of HE data validation checklist, Part I;
  - 6.3.2.2 circle “A, H16” in block 3b of the HE data validation checklist, Part I;
  - 6.3.2.3 record the qualifier flag reason and code combination “A, H16” next to the results of all affected samples, on Form I; and
  - 6.3.2.4 record the time elapsed (to the nearest minute) between completion of the daily calibration and completion of the

affected sample analyses quantitated under the daily calibration, in block 2b of HE validation checklist, Part I.

#### 6.4 Verify Method-Blank Results

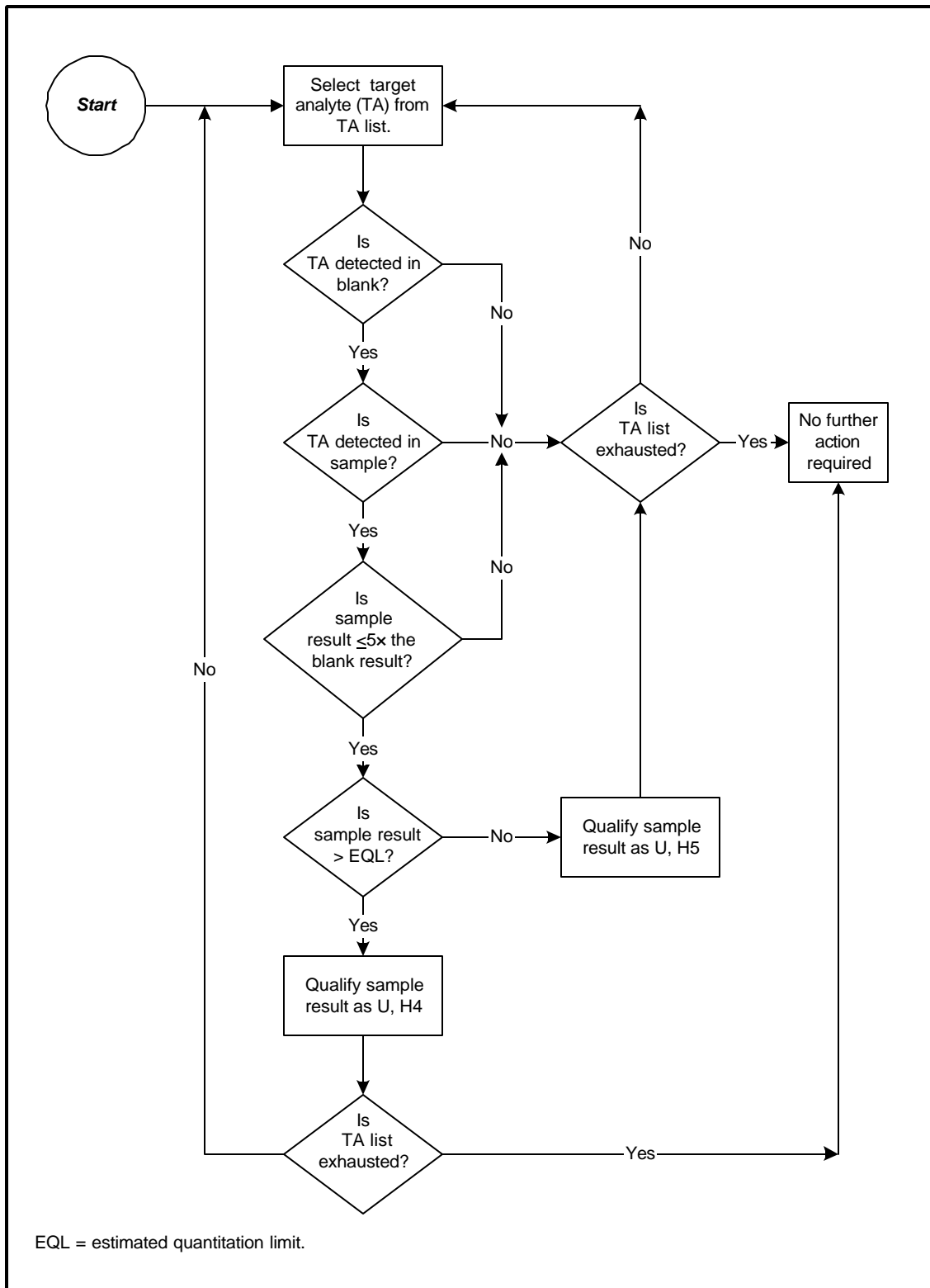
**Note:** The data validator must compare method-blank results to the contractually required EQLs.

**Note:** If additional validation forms are needed to record validation data for more than one blank, copy the appropriate forms and use the copies for the additional information.

- 6.4.1 If a method blank *was analyzed* for each sample matrix and/or analytical batch,
  - 6.4.1.1 record “Y” in block 1a of HE data validation checklist, Part IIa;
  - 6.4.1.2 record “n/a” in block 1c of HE data validation checklist, Part IIa; and
  - 6.4.1.3 go to Section 6.4.3.
- 6.4.2 If a method blank *was not analyzed* for each sample matrix and/or analytical batch,
  - 6.4.2.1 record “N” in block 1a of the HE data validation checklist, Part IIa;
  - 6.4.2.2 circle “A, H5a” in block 1b of the HE data validation checklist, Part IIa;
  - 6.4.2.3 record the qualifier flag and reason code combination “A, H5a” next to the results of all samples for which a method blank was not analyzed, on Form I;
  - 6.4.2.4 record the matrices and/or analytical batches that did not include a method-blank analysis, in block 1c of the HE data validation checklist, Part IIa; and
  - 6.4.2.5 go to Section 6.5, Verify Laboratory Control Sample Results.
- 6.4.3 If *no* target analytes were detected in the method blank,
  - 6.4.3.1 record “N” in blocks 2a and 3a of the HE data validation checklist, Part IIb;
  - 6.4.3.2 record “n/a” in blocks 2c, 2d, 3c, and 3d of the HE data validation checklist, Part IIb; and
  - 6.4.3.3 go to Section 6.5,. Verifying LCS Results.

- 6.4.4 If the concentration of any sample analyte that *was detected* in a method blank is *not* greater than EQL or is *not* less than or equal to five times the concentration in the blank,
  - 6.4.4.1 record “N” in block 2a of the HE data validation checklist, Part IIb;
  - 6.4.4.2 record “n/a” in blocks 2c and 2d of the HE data validation checklist, Part IIb; and
  - 6.4.4.3 go to Section 6.4.6.
- 6.4.5 If the concentration in a sample of any sample analyte that *was detected* in a method blank is greater than EQL *and* less than or equal to five times the concentration in the blank,
  - 6.4.5.1 record “Y” in block 2a of the HE data validation checklist, Part IIb;
  - 6.4.5.2 circle “U, H4” in block 2b of the HE data validation checklist, Part IIb;
  - 6.4.5.3 record the qualifier flag and reason code combination “U, H4” next to the result for each affected target analyte, on Form I;
  - 6.4.5.4 record the samples that have been qualified “U” and the analytes that were detected in the blank, in block 2c of the HE data validation checklist, Part IIb; and
  - 6.4.5.5 record the analyte names and their method-blank concentrations for all HE analytes detected in the blank, in block 2d of the HE data validation checklist, Part IIb.
- 6.4.6 If the concentration of any sample analyte that *was detected* in a method blank is *not* less than or equal to five times the concentration in the blank,
  - 6.4.6.1 record “N” in block 3a of the HE data validation checklist, Part IIb;
  - 6.4.6.2 record “n/a” in blocks 3c and 3d of the HE data validation checklist, Part IIb; and
  - 6.4.6.3 go to Section 6.5, Verify Laboratory Control Sample Results.

- 6.4.7 If the concentration of any sample analyte that *was detected* in a blank is less than EQL *and* less than five times the concentration in the blank,
- 6.4.7.1 record “Y” in block 3a of the HE data validation checklist, Part IIb;
  - 6.4.7.2 circle “U, H5” in block 3b of the HE data validation checklist, Part IIb;
  - 6.4.7.3 record the qualifier flag and reason code combination “U, H5” next to the result for each affected target analyte, on Form I;
  - 6.4.7.4 record the samples that have been qualified “U” and the analytes that were detected in the blank in block 3c of the HE data validation checklist, Part IIb; and
  - 6.4.7.5 record the analyte names and their blank concentrations for all HE analytes detected in the blank in block 3d of the HE data validation checklist, Part IIb.
- 6.4.8 Use the logic diagram in Figure 6.4-1 to determine which, if any, LANL qualifier flags and reason codes **validator** must assign to the sample results for noncompliant method blanks.



**Figure 6.4-1.** Applying LANL qualifier flags and reason codes to the sample results for noncompliant method blanks.

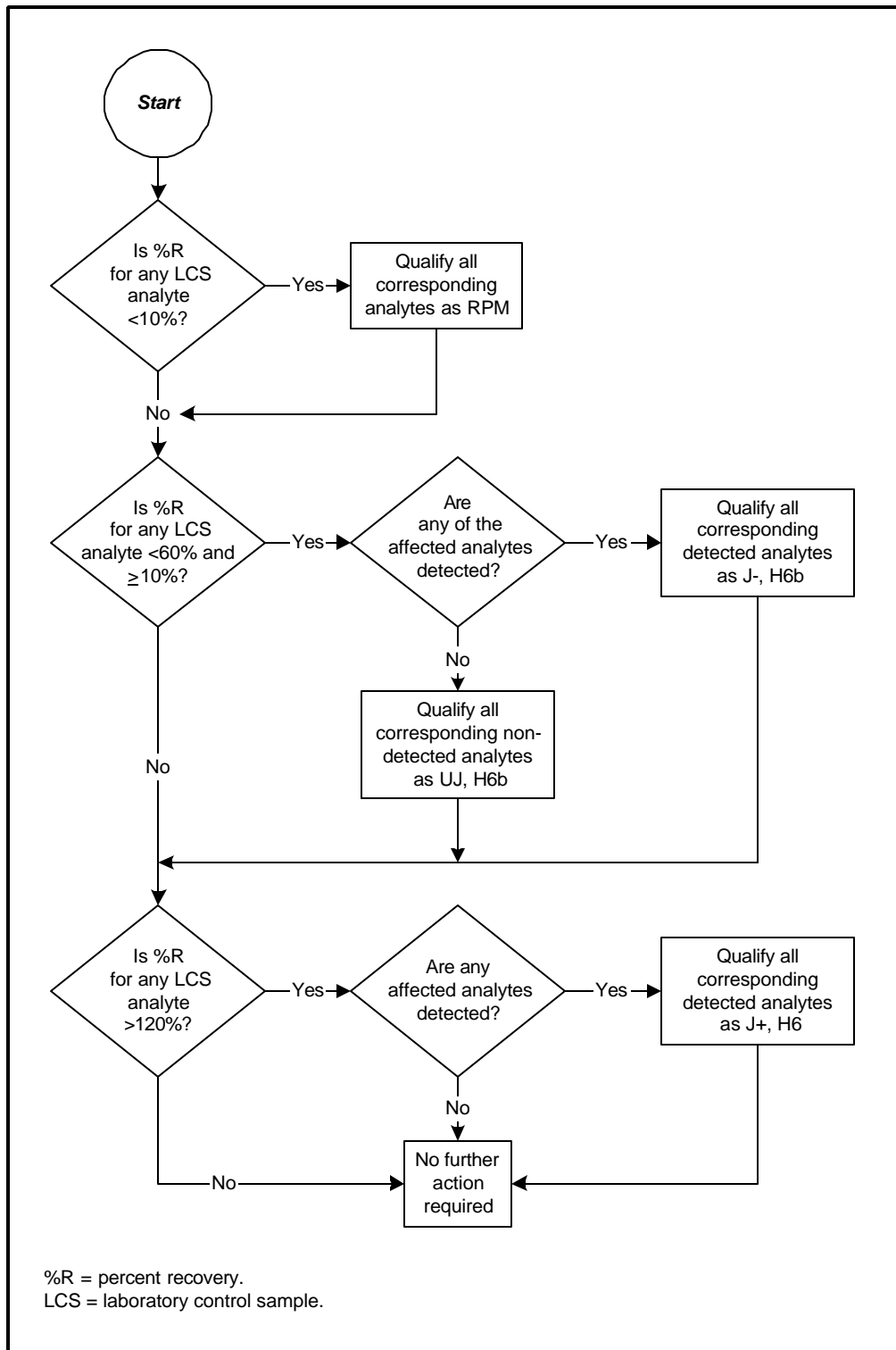
## 6.5 Verify Laboratory Control Sample Results

**Note:** The LCS shall contain at least seven target analytes.

- 6.5.1 If the percent recovery (%R) for any LCS analyte is *not reported* for a sample,
  - 6.5.1.1 record “Y” in block 1a of the HE data validation checklist, Part III;
  - 6.5.1.2 circle “A, H12” in block 1b of the HE data validation checklist, Part III;
  - 6.5.1.3 record the qualifier flag and reason code combination “A, H12” next to all results of each sample that do not contain the required analyte results, on Form I; and
  - 6.5.1.4 record the affected samples in block 1c of the HE data validation checklist, Part IV.
- 6.5.2 If *all* LCS analytes are greater than 10%R,
  - 6.5.2.1 record “N” in block 2a of the HE data validation checklist, Part III.
  - 6.5.2.2 record “n/a” in blocks 2c and 2d of the HE data validation checklist, Part III; and
  - 6.5.2.3 go to Section 6.5.4.
- 6.5.3 If *any* LCS analyte is less than 10%R,
  - 6.5.3.1 record “Y” in block 2a of the HE data validation checklist, Part III;
  - 6.5.3.2 circle “RPM, H6d” in block 2b of the HE data validation checklist, Part III;
  - 6.5.3.3 record the qualifier flag and reason code combination “RPM, H6d” next to all analytes that correspond to the noncompliant LCS analyte results, on Form I;
  - 6.5.3.4 record the affected samples in block 2c of the HE data validation checklist, Part III; and
  - 6.5.3.5 record the %R values of the affected analytes in block 2d of the HE data validation checklist, Part III.
- 6.5.4 If *all* LCS analytes are greater than 60%R,
  - 6.5.4.1 record “N” in block 3a of the HE data validation checklist, Part III;

- 6.5.4.2 record “n/a” in blocks 3c and 3d of the HE data validation checklist, Part III; and
- 6.5.4.3 go to Section 6.5.6.
- 6.5.5 If *any* LCS analyte is less than 60%R but greater than 10%R,
  - 6.5.5.1 record “Y” in block 3a of the HE data validation checklist, Part III,
  - 6.5.5.2 and for each *detected* sample result that corresponds to a noncompliant LCS analyte,
    - 1) circle “J-, H6b” in block 3b of the HE data validation checklist, Part III;
    - 2) record the qualifier flag and reason code combination “J-, H6b” next to all affected sample results, on Form I;
    - 3) record the affected sample analytes in block 3c of the HE data validation checklist, Part III; and
    - 4) record the %R values of the affected analytes, in block 3d of the HE data validation checklist, Part III;
  - 6.5.5.3 and for each *nondetected* sample result that corresponds to a noncompliant LCS analyte,
    - 1) circle “UJ, H6b” in block 3b of the HE data validation checklist, Part III;
    - 2) record the qualifier flag and reason code combination “UJ, H6b” next to all sample results that correspond to the noncompliant LCS analyte results, on Form I;
    - 3) record which analytes are affected in block 3c of the HE data validation checklist, Part III; and
    - 4) record the %R values of the affected analytes in block 3d of the HE data validation checklist, Part III.
- 6.5.6 If *any* LCS analyte is greater than 120%R and the sample analyte is *not detected*,
  - 6.5.6.1 record “N” in block 4a of the HE data validation checklist, Part III;
  - 6.5.6.2 record “n/a” in blocks 4c, and 4d of the HE data validation checklist, Part III; and
  - 6.5.6.3 go to Section 6.6, Verify Surrogate Recoveries.

- 6.5.7 If *any* LCS analyte is greater than 120%R and the sample analyte is *detected*,
- 6.5.7.1 record “Y” in block 4a of the HE data validation checklist, Part III;
  - 6.5.7.2 circle “J+, H6” in block 4b of the HE data validation checklist, Part III;
  - 6.5.7.3 record the qualifier flag and reason code combination “J+, H6” next to all sample results that correspond to the noncompliant LCS analyte results, on Form I;
  - 6.5.7.4 record the affected analytes in block 4c of the HE data validation checklist, Part III; and
  - 6.5.7.5 record the %R values of the affected analytes in block 4d of the HE data validation checklist, Part III.
- 6.5.8 Use the logic diagram in Figure 6.5-1 to determine which, if any, laboratory LANL qualifier flags the and reason codes the **validator** must assign to the sample results for noncompliant sample results.



**Figure 6.5-1.** Applying LANL qualifiers and reason codes to the sample results for noncompliant LCSs.

## 6.6 Verify Surrogate Recoveries

**Note:** Surrogate %R values that are outside the acceptance range listed in Table 6.6-1 as a result of sample dilution used to render target analytes quantifiable are not subject to the validation-acceptance criteria presented in this section (Section 6.6).

6.6.1 If the %R value for 3,4-dinitrotoluene listed in Table 6.6-1 *is reported* for all samples,

6.6.1.1 record “N” in block 1a of the HE data validation checklist, Part IV.

6.6.1.2 record “n/a” in block 1c of the HE data validation checklist, Part IV; and

6.6.1.3 go to Section 6.6.3.

**Note:** Inclusion of the surrogate 4-nitroaniline is not a requirement.

**Table 6.6-1.  
HE Surrogate and Recovery Acceptance Ranges**

Surrogate	Soil Matrix Acceptance Range (%R)	Water Matrix Acceptance Range (%R)
3,4-Dinitrotoluene (required)	50–160	50–160
4-Nitroaniline (optional)	50–160	50–160

6.6.2 If the %R value for 3,4-dinitrotoluene (see Table 6.6-1) is *not reported* for a sample,

6.6.2.1 record “Y” in block 1a of the HE data validation checklist, Part IV;

6.6.2.2 circle “A, H3g” in block 1b of the HE data validation checklist, Part IV;

6.6.2.3 record the qualifier flag and reason code combination “A, H3g” next to all results of each sample that does not contain the required surrogates, on Form I;

6.6.2.4 record the affected samples in block 1c of the HE data validation checklist, Part I.

6.6.3 For *all* reported surrogate %R values, compare the reported recovery to the corresponding recovery value in Table 6.6-1.

6.6.4 For each sample, if *no* surrogate is less than 10%R,

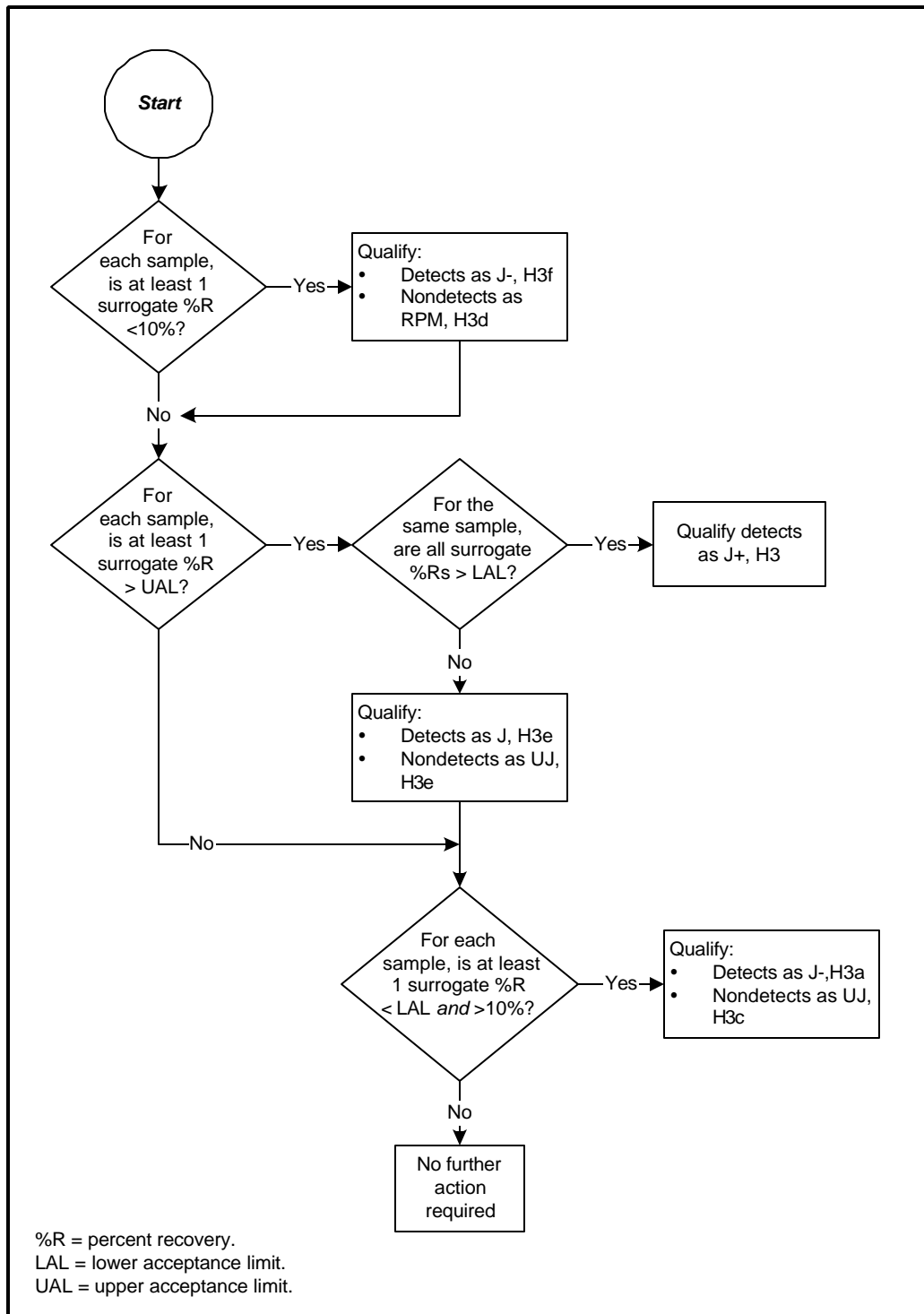
6.6.4.1 record “N” in block 2a of the HE data validation checklist, Part IV;

- 6.6.4.2 record “n/a” in block 2c and 2d of the HE data validation checklist, Part IV; and
- 6.6.4.3 go to Section 6.6.6.
- 6.6.5 For each sample, if at least *one* reported surrogate is less than 10%R,
  - 6.6.5.1 record “Y” in block 2a of the HE data validation checklist, Part IV
  - 6.6.5.2 and for target analytes that *are detected* in the affected sample,
    - 1) circle “J-, H3f” in block 2b of the HE data validation checklist, Part IV;
    - 2) record the qualifier flag and reason code combination “J-, H3f” next to the result of each detected target analyte, on Form I;
    - 3) record the noncompliant surrogates and samples in block 2c of the HE data validation checklist, Part IV; and
    - 4) record the %R value that corresponds to the identified noncompliant surrogates in block 2d of the HE data validation checklist, Part IV;
  - 6.6.5.3 and for target analytes that *are not detected* in the affected sample,
    - 1) circle “RPM, H3d” in block 2b of the HE data validation checklist, Part IV;
    - 2) record the qualifier flag and reason code combination “RPM, H3d” next to the result of each nondetected target analyte, on Form I;
    - 3) record the noncompliant surrogates and samples in block 2c of the HE data validation checklist, Part IV; and
    - 4) record the %R values that correspond to the identified noncompliant surrogates in block 2d of the HE data validation checklist, Part IV.
- 6.6.6 For each sample, if *no* surrogate %R value falls outside its acceptance limit,
  - 6.6.6.1 record “N” in block 3a of the HE data validation checklist, Part IV;

- 6.6.6.2 record "n/a" in blocks 3c and 3d of the HE data validation checklist, Part IV; and
- 6.6.6.3 go to Section 6.6.8.
- 6.6.7 For each sample, if at least *one* surrogate %R value exceeds its UAL, *and* if no surrogate %R values are less than their LALs,
  - 6.6.7.1 record "Y" in block 3a of the HE data validation checklist, Part IV
  - 6.6.7.2 and if any target analyte *is detected* in the affected sample,
    - 1) circle "J+, H3" in block 3b of the HE data validation checklist, Part IV;
    - 2) record the qualifier flag and reason code combination "J+, H3" next to the result of each detected target analyte, on Form I;
    - 3) record the noncompliant surrogates and samples in block 3c of the HE data validation checklist, Part IV; and
    - 4) record the %R values of the noncompliant surrogates in block 3d of the HE data validation checklist, Part IV;
  - 6.6.7.3 and if *no* target analytes are detected in the affected sample,
    - 1) record "n/a" in blocks 3a, 3c, and 3d of the HE data validation checklist, Part IV;
  - 6.6.7.4 or if *at least one* surrogate %R value is less than the LAL, record
    - 1) "N" in block 3a of the HE data validation checklist, Part IV and
    - 2) "n/a" in blocks 3c and 3d and of the HE data validation checklist, Part IV.
- 6.6.8 If *at least one* surrogate *does not* fall outside *each* acceptance limit,
  - 6.6.8.1 record "N" in block 4a of the HE data validation checklist, Part IV;
  - 6.6.8.2 record "n/a" in block 4c and 4d of the HE data validation checklist, Part IV; and
  - 6.6.8.3 go to Section 6.7, Verify Holding Times.

- 6.6.9 For each sample, if *at least one* surrogate %R value is greater than UAL *and at least one* surrogate %R value is less than LAL,
- 6.6.9.1 record “Y” in block 4a of the HE data validation checklist, Part IV
- 6.6.9.2 and if any analyte *is detected* in the affected sample,
- 1) circle “J, H3e” in block 4b of the HE data validation checklist, Part IV;
  - 2) record the qualifier flag and reason code combination “J, H3e” next to the result of each detected target analyte, on Form I;
  - 3) record the noncompliant surrogates and samples in block 4c of the HE data validation checklist, Part IV; and
  - 4) record the %R values that correspond to the identified noncompliant surrogates in block 4d of the HE data validation checklist, Part IV;
- 6.6.9.3 and if any target analyte is *not detected* in the affected sample,
- 1) circle “UJ, H3e” in block 4b of the HE data validation checklist, Part IV;
  - 2) record the qualifier flag and reason code combination “UJ, H3e” next to the result of each undetected target analyte, on Form I;
  - 3) record the noncompliant surrogates and samples in block 4c of the HE data validation checklist, Part IV; and
  - 4) record the %R values that correspond to the identified noncompliant surrogates in block 4d of the HE data validation checklist, Part IV.
- 6.6.10 If *at least one* surrogate is *not* less than LAL,
- 6.6.10.1 record “N” in block 5a of the HE data validation checklist, Part IV.
- 6.6.10.2 record “n/a” in blocks 5c and 5d of the HE data validation checklist, Part IV; and
- 6.6.10.3 go to Section 6.7, Verify Holding Times.

- 6.6.11 If at least one surrogate %R value is less than its LAL but is greater than 10%R,
- 6.6.11.1 record “Y” in block 5a of the HE data validation checklist, Part IV
- 6.6.11.2 and if any target analyte *is detected* in the sample,
- 1) circle “J-,H3a” in block 5b of the HE data validation checklist, Part IV;
  - 2) record the qualifier flag and reason code combination “J, H3a” next to the result of each detected target analyte, on Form I;
  - 3) record the noncompliant surrogates and samples in block 5c of the HE data validation checklist, Part IV; and
  - 4) record the %R values that correspond to the identified noncompliant surrogates in block 5d of the HE data validation checklist, Part IV;
- 6.6.11.3 and if any target analyte *is not detected* in the affected sample,
- 1) circle “UJ, H3c” in block 5b of the HE data validation checklist, Part IV;
  - 2) record the qualifier flag and reason code combination “UJ, H3c” next to the result of each undetected target analyte, on Form I;
  - 3) record the noncompliant surrogates and samples in block 5c of the HE data validation checklist, Part IV; and
  - 4) record the %R values that correspond to the identified noncompliant surrogates in block 5d of the HE data validation checklist, Part IV.
- 6.6.12 Use the logic diagram in Figure 6.6-1 to determine which, if any, LANL qualifier flags the and reason codes **validator** must assign to the sample results for noncompliant surrogate compounds.



**Figure 6.6-1.** Applying LANL qualifier flags and reason codes to the sample results for noncompliant surrogate compounds.

## 6.7 Verify Holding Time

### 6.7.1 Verify Extraction Holding-Time Results

6.7.1.1 If *all* samples were *prepared* within the extraction holding time (see Table 6.7-1),

- 1) record “Y” in block 1a of the HE data validation checklist, Part V;
- 2) record “n/a” in blocks 1c and 1d of the HE data validation checklist, Part V; and
- 3) go to Section 6.7.2.

**Table 6.7-1.**  
**Holding Time Acceptance Criteria\***

<b>Sample Matrix</b>	<b>Extraction Holding Time (days)</b>	<b>Analysis Holding Time (days)</b>
Soil	14	40
Water	7	40
* Applicable storage conditions are listed in the current SOW for analytical services.		

6.7.1.2 If *any* samples were *not prepared* within the extraction holding time (see Table 6.7-1),

- 1) record “N” in block 1a of the HE data validation checklist, Part V;
- 2) circle “PM, H9” in block 1b of the HE data validation checklist, Part V;
- 3) record the qualifier flag and reason code combination “PM, H9” next to the result of each affected target analyte, on Form I;
- 4) record the affected samples in block 1c of the HE data validation checklist, Part V; and
- 5) record the number of days by which the holding time was exceeded, for each sample that exceeded its holding time, in block 1d of the HE data validation checklist, Part V.

## 6.7.2 Verify Analytical Holding-Time Results

6.7.2.1 If *all* samples were analyzed within the analytical holding time (see Table 6.7-1),

- 1) record “Y” in block 1a of the HE data validation checklist, Part V;
- 2) record “n/a” in blocks 1c and 1d of the HE data validation checklist, Part V; and
- 3) go to Section 6.8, Assemble the Validation Data Record Package.

6.7.2.2 If *any* samples were *not analyzed* within the analytical holding time (see Table 6.7-1),

- 1) record “N” in block 2a of the HE data validation checklist, Part V;
- 2) circle “PM, H9” in block 2b of the HE data validation checklist, Part V;
- 3) record the qualifier flag and reason code combination “PM, H9” next to the result of each affected target analyte, on Form I;
- 4) record the samples that are affected in block 2c of the HE data validation checklist, Part V; and
- 5) record the number of days by which the holding time was exceeded, for each sample that exceeded its holding time, in block 2d of the HE data validation checklist, Part V.

6.8 Assemble the validation data record package to include the following items in the order they are listed below:

- 6.8.1 completed, signed, and dated data validation cover sheet;
- 6.8.2 the HE data validation checklists completed in Sections 6.2 through 6.7;
- 6.8.3 photocopies of the completed forms (Form I) on which the validator recorded data validation qualifier flags and reason codes;
- 6.8.4 a photocopy of the data record package case narrative; and
- 6.8.5 photocopies of the data record package QC forms (assemble in order by QC forms).

6.9 Submit the validation data record package to the SMO, in accordance with ER-SOP-15.09.

## **7.0 REFERENCES**

The following documents are cited within this procedure:

EPA (US Environmental Protection Agency), February 1994. "US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," Publication 9240.1-05, EPA-540/R-94/012, Office of Solid Waste and Emergency Response, Washington, DC.

ER-SOP-15.09, Chain of Custody for Analytical Data Packages

LANL (Los Alamos National Laboratory), July 1995. "Environmental Restoration Project Statement of Work for Analytical Services," Revision 2, RFP Number 9-SX1-Q4257, Los Alamos National Laboratory, Los Alamos, New Mexico.

QP-2.2, Personnel Orientation and Training

QP-4.2, Standard Operating Procedure Development

## **8.0 RECORDS**

Although no records will be submitted to the Records Processing Facility (RPF) in the course of completing this procedure, the items identified in Section 6.11 will be a part of the data record package submitted to the RPF from the SMO in accordance with ER-SOP-15.09.

## **9.0 ATTACHMENTS**

The document user may employ documentation formats different from those attached to/named in this procedure—as long as the substituted formats in use provide, as a minimum, the information required in the official forms developed by the procedure.

Attachment A: High Explosives Data Validation Qualifier Flags (1 page)

Attachment B: High Explosives Data Validation Reason Codes (2 pages)

Attachment C: Data Validation Cover Sheet (1 page)

Attachment D: High Explosives Data Validation Checklist forms, Part I through Part V (5 pages)

## High Explosives Data Validation Qualifier Flags

- A The contractually required supporting documentation for this datum is absent.
- U The analyte is classified as “not detected.”
- J The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual.
- J+ The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual with a potential positive bias.
- J- The analyte is classified as “detected” but the reported concentration value is expected to be more uncertain than usual with a potential negative bias.
- UJ The analyte is classified as “not detected” with an expectation that the reported result is more uncertain than usual.
- RPM The reported sample result is classified as “rejected” due to serious noncompliances regarding QC acceptance criteria. The presence or absence of the analyte cannot be verified based on routine validation alone.
- PM Manual review of raw data is recommended to determine if the observed non-compliances with quality acceptance criteria adversely impacts data use.
- Note:** A “PM” qualifier flag indicates that a manual review should be conducted if the datum that is qualified with the “PM” is important to the data user. In addition, "PM" means that a decision must be made by the project manager/delegee regarding the need for further review of the data. This review should include some consideration of potential impact that could result from using the "PM" qualified data.

## High Explosives Data Validation Reason Codes

- H3 The surrogate percent recovery is greater than the UAL, which indicates the potential for a high bias in the results and the potential for false positive results
- H3a The surrogate percent recovery is less than the LAL but greater than 10%R, which indicates the potential for a low bias in the detected results.
- H3c The reporting limit is approximated for nondetects because a surrogate percent recovery is lower than the LAL but greater than or equal to 10%R, which indicates an increased potential for false negative results.
- H3d The surrogate is less than 10%R and the result is a nondetect, which indicates significant potential for false negative results.
- H3e At least one surrogate percent recovery exceeds its upper UAL and at least one surrogate is less than its LAL, which indicates a greater than normal degree of uncertainty in the data.
- H3f At least one surrogate is less than 10%R and the sample result is a detect, which indicates the potential for a severely low bias in the results.
- H3g Required surrogate information is missing. Data may not be acceptable for use.
- H4 The sample result is greater than the EQL and less than five times the concentration of the related analyte in the blank, which indicates that the reported detection is considered indistinguishable from blank contamination.
- H5 The sample result is less than the EQL and less than five times the concentration of the analyte in the method blank, which indicates the reported detection is considered indistinguishable from contamination in the blank.
- H5a Method-blank data is missing, or method blank was not analyzed. Data may not be acceptable for use.
- H6 The recovery of the LCS analyte is greater than the UAL, which indicates the potential for high bias in the results and for false positive results.
- H6b The of the LCS analyte percent recovery is less than the LAL and greater than or equal to 10%R, which indicates (1) the reporting limit is approximate and probably biased low for nondetected results, and (2) that detected results likely are biased low.

- H6d The result is a nondetect and the %R value of surrogates or the analyte in the LCS is less than 10%R, which indicates a greatly increased potential for false negative results.
- H9 The holding time is exceeded. The data user should conduct a technical evaluation of the data of interest with respect to the effects of exceeding the holding time. Factors to consider include how long the holding time was exceeded, sample preservation, sample storage practices, use of the data, levels of contamination found in the sample, and the physical, chemical, and biological stability of the target analytes in the sample matrix.
- H12 Required LCS data are missing. The LCS analyte recoveries could not be evaluated. Data may not be acceptable for use.
- H14a Insufficient sample volume was received for a matrix spike and/or a matrix spike duplicate analysis.  
**Note:** A matrix spike duplicate is not appropriate for all analyses.
- H14b The matrix spike and/or the matrix spike duplicate analyses were not performed on a sample associated with a LANL request number.  
**Note:** A matrix spike duplicate is not appropriate for all analyses.
- H14c The matrix spike and/or the matrix spike duplicate were analyzed on a sample associated with a different LANL request number but no summary was included.  
**Note:** A matrix spike duplicate is not appropriate for all analyses.
- H15 Because the sample was damaged, lost, or of insufficient quantity, the laboratory was unable to analyze it.
- H16 Required calibration information is missing or samples were analyzed on an expired calibration. Data may not be acceptable for use.

## Data Validation Cover Sheet

### Section I.

Request Number: \_\_\_\_\_ Validation Date: \_\_\_\_\_ Lab Code: \_\_\_\_\_

Contract Laboratory Name: \_\_\_\_\_

Validator: \_\_\_\_\_ Organization: \_\_\_\_\_

Analytical Suite (check all that apply): ☐ Volatile Organics ☐ High Explosives  
☐ Semivolatile Organics ☐ Inorganics  
☐ Organochlorine Pesticides/Polychlorinated Biphenyls ☐ Radiochemistry

Other (describe): \_\_\_\_\_

### Section II. Completeness Check

Yes	No	n/a	(check one)	Yes	No	n/a	(check one)
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1. Chain-of-custody form(s)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	6. Raw/BSS data
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2. Case narrative	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7. Quality control forms
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3. Sample result forms	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	8. Quantitation reports
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. Sample chromatograms	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9. TICs forms
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	5. Standard chromatograms	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	10. TICs mass spectra

Identify any samples in the assigned Request Number that are missing:

\_\_\_\_\_

\_\_\_\_\_

Comments/problems noted (include information about samples for further information submitted to the contract laboratory and agreed upon date of resolution and contract laboratory point of contact):

(Attach additional comment sheets as necessary)

Validator's signature: \_\_\_\_\_ Date: \_\_\_\_\_

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## High Explosives Data Validation Checklist

### Part I. Instrument Calibrations

Criteria	Criterion true? (y, n, or n/a)	Action if “criterion true?” = no	Actual time lapse
Was the initial calibration completed?	1a.	1b. In block 1c, record the actual time lapse.	1c.
Was the daily calibration check performed at the beginning of each 24-hour analysis period following the analysis of the instrument performance check sample and before the analysis of blanks and samples? <b>Attention:</b> A daily calibration check is not required if all samples are analyzed within 24 hours of initial calibration—record “n/a” in blocks 2a and 2c.	2a.	2b. In block 2c, record the actual time lapse.	2c.
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## High Explosives Data Validation Checklist

### Part IIa. Method Blanks Validation Criteria

Criterion	Criterion true? (yes or no)	Action if "criterion true?" = no Assign qualifier ...	List affected matrices or batches.
Was a method blank analyzed for each sample matrix and/or batch?	1a.	1b. "A" for any missing documentation. In block 1c, record all sample matrices and/or analytical batches that did not include a method blank.	1c.

### Part IIb. Method Blanks Validation Criteria (continued)

Criteria	Criterion true? (yes or no)	Action if "criterion true?" = no Assign qualifier & reason code...	List detected blank analyte(s) and affected samples.	Analyte concentration (mg/kg)
Is a target analyte detected in both the method blank and sample <b>AND</b> is the sample result > the EQL and = 5 times the method blank concentration?	2a.	2b. "U, H4" to the sample analyte(s) in question (in blocks 2c and 2d).	2c.	2d.
Is a target analyte detected in both the method blank and sample <b>AND</b> is the sample result < the EQL <b>AND</b> is the sample result = 5 times the method blank concentration?	3a.	3b. "U, H5" to the sample analyte(s) in question (in blocks 3c and 3d).	3c.	3d.

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## High Explosives Data Validation Checklist

### Part III. Laboratory Control Sample Validation Criteria

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = yes Identify noncompliant LCS analytes <u>and</u> assign qualifier & reason code...	List all noncompliant analytes and samples.	Percent recovery
Are any required analyte percent recoveries <u>not</u> reported?	1a.	1b. "A" for any missing documentation. In block 1c, record <ul style="list-style-type: none"> <li>noncompliant analytes and</li> <li>all affected samples.</li> </ul>	1c.	1d. n/a
Are any analyte percent recoveries <10%?	2a.	2b. "RPM, H6d" to all affected analytes. In block 2c, record <ul style="list-style-type: none"> <li>noncompliant analytes and</li> <li>all affected sample analytes.</li> </ul> In block 2d, record the percent recoveries of all affected analytes.	2c.	2d.
Are any analyte recoveries < 60%  <b>BUT</b>  ≥ 10%?	3a.	3b. "J-, H6b" to all <u>detected</u> sample analytes and "UJ, H6b" to all <u>nondetected</u> sample analytes. In block 3c, record <ul style="list-style-type: none"> <li>noncompliant analytes and</li> <li>all affected sample analytes.</li> </ul> In block 3d, record the percent recoveries of all affected analytes.	3c.	3d.
Are any analyte recoveries > 120%?	4a.	4b. "J+, H6" to all <u>detected</u> sample analytes In block 4c, record <ul style="list-style-type: none"> <li>noncompliant analytes and</li> <li>all affected sample analytes.</li> </ul> In block 4d, record the percent recoveries of all affected analytes.	4c.	4d.
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## High Explosives Data Validation Checklist

### Required HE Surrogate Compounds and Recovery Acceptance Ranges

Surrogate name	Soil matrix recovery acceptance range	Water matrix recovery acceptance range
3,4-Dinitrotoluene (required)	50%–160%	50%–160%
4-Nitroaniline (optional)	50%–160%	50%–160%

### Part IV. Surrogate Validation Criteria

Criteria	Criterion true? (yes/no)	Action if “criterion true?” = yes Identify noncompliant surrogates <u>and</u> assign qualifier & reason code...	List all noncompliant surrogates and samples.	Percent recovery
Are any required surrogate percent recoveries <u>not</u> reported?	1a.	1b. “A” for any missing documentation. In block 1c, record <ul style="list-style-type: none"> <li>• noncompliant surrogates and</li> <li>• all affected samples.</li> </ul>		1d. n/a
Is at least one surrogate recovery <10%?	2a.	2b. “J-, H3f” to all <u>detected</u> sample analytes and “RPM, H3d” to all <u>nondetected</u> sample analytes.	2c.	2d.
Is at least one surrogate percent recovery > UAL <b>AND</b> no surrogate percent recoveries < LAL?	3a.	3b. “J+, H3e” to all detected sample analytes.	3c.	3d.
Is at least one surrogate percent recovery > UAL <b>AND</b> at least one surrogate percent recovery <LAL?	4a.	4b. “J, H3e” to all <u>detected</u> sample analytes and “UJ, H3e” to all <u>nondetected</u> sample analytes.	4c.	4d.
Is at least one surrogate percent recovery <u>AND</u> <u>the same</u> surrogate percent recovery > 10%?	5a.	5b. “J-, H3a” to all <u>detected</u> sample analytes and “UJ-, H3c” to all <u>nondetected</u> sample analytes.	5c.	5d.

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## High Explosives Data Validation Checklist

### Part V. Holding Time Validation Criteria

Criteria	Criterion true? (yes/no)	Action if "criterion true?" = no Assign qualifier & reason code...	List samples for which holding times were exceeded.	List the number of days by which holding times were exceeded.
Was each sample prepared within its required extraction holding time?	1a.	1b. "PM, H9" to all analytes in affected samples.	1c.	1d.
Was each sample analyzed within its required analytical holding time?	2a.	2b. "PM, H9" to all analytes in affected samples.	2c.	2d.
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